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(54) Title: METHOD AND DEVICE FOR BIOGAS UPGRADING AND HYDROGEN PRODUCTION FROM ANAEROBIC FERMENTATION OF BIOLOGICAL MATERIAL

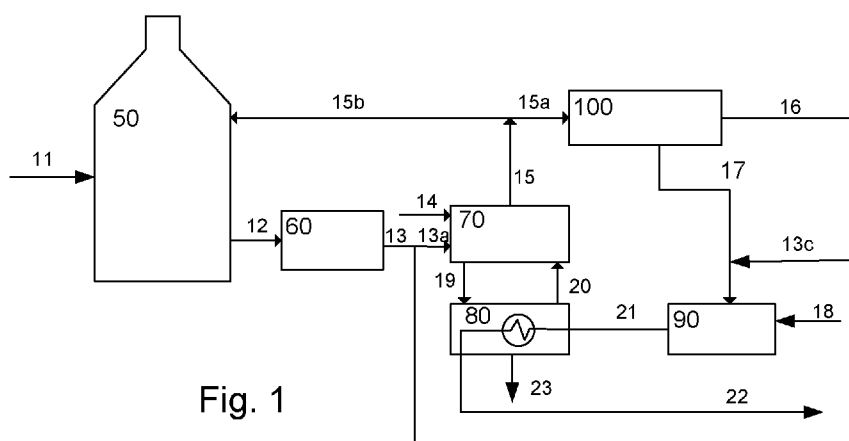


Fig. 1

(57) Abstract: Method and device for biogas upgrading and hydrogen production from anaerobic fermentation of biological material under production of energy rich gases selected among methane (24) and hydrogen (16, 26) or a combination thereof. The method comprises addition of hydrogen gas to a fermentation step (50) to enhance the methane:CO₂ ratio in the raw biogas (12) produced. At least part of the raw biogas is subjected to a step of sorption enhanced reforming (70) without prior separation of CO₂, using CaO as an absorbent to capture CO₂ from the raw biogas as well as CO₂ released in the reforming reaction. CaO is regenerated in an endothermic reaction (80) using heat at least partially provided, directly or indirectly, by the bio-gas to be upgraded, thereby producing substantially pure hydrogen (15) and substantially pure CO₂ (23).



Method and device for biogas upgrading and hydrogen production from anaerobic fermentation of biological material

The present invention concerns a method for anaerobic fermentation of biological material under production of energy rich gases selected among methane and hydrogen as disclosed by the preamble of claim 1 and a device for conducting the method as disclosed by the preamble of claim 12.

Background

Biogas can be produced by anaerobic digestion (AD) or fermentation of biodegradable materials, such as manure, wastewater and sewage sludge, municipal waste, green waste, plant material, and crops. Biogas consists mainly of methane (CH₄) and carbon dioxide (CO₂), and small amounts of hydrogen sulphide (H₂S), moisture, and siloxanes.

If the anaerobically produced biogas is to be used as a transportation fuel (vehicle grade), it first has to be upgraded to remove impurities and increase its calorific value (heating value). This upgrading step includes drying, desulfurization, and CO₂-removal. The CO₂-separation is normally carried out by water scrubbing, physical or chemical absorption using organic solvents, pressure swing adsorption, or by permeation using membranes, depending on the location and size of the system.

In a presentation made at Group Exhibit 2015 Hydrogen Fuel Cells Batteries, April 14, 2015), as also documented in *Energia Procedia* 63 (2014) 279-285, published by Elsevier Ltd., a plant for so-called bio-ZEG methane was presented and discussed, including the use of solid oxide fuel cells and use of CaO as a CO₂ scavenger. There was, however, no mention of using raw CO₂ containing biogas as a source feed for such a process.

US 2015 0284247 A1 teaches an apparatus for producing high-purity gas which includes a column configured for sorption-enhanced reaction (SE-SMR) for removing a by-product through a catalyst reaction. The column is divided into a plurality of sections, the sections having decreasing proportions of catalyst and increasing proportions of an absorbent.

US 2013 011326 A1 and DE 19 946 381 A1 are other publications in this technical field.

However, one of the main drawbacks and challenges with biogas is the requirement for upgrading to bio-methane, with a quality for use as fuel for (bio) gas vehicles, or as a source for hydrogen fuel cell electric vehicles (FCEVs). Biogas from food waste (or other sources such as manure and wastewater) treatment facilities typically consist of 55-65% CH₄ and 35-45% CO₂. Upgrading (CO₂

removal) consumes energy and adds significant costs to the overall system (Luo and Angelidaki, 2012). Hence, there is a need to find new, more efficient, and less costly methods for upgrading anaerobically produced biogas for example used directly as fuel in vehicles, and as a source for hydrogen production used in hydrogen fuel cell electric vehicles.

- 5 There is still, however, a need for further improvements in this area to make fuel produced by digestion of organic waste competitive as vehicle fuel.

This can to some extent be achieved by reduction of the CO₂ content and enhancement of the methane content, by adding hydrogen to the anaerobic digestion (AD) process, thereby increasing the methane content to about 80%

10 Objectives

The main objective of the present invention is thus to develop a new and cost-efficient process that allows production of vehicle grade fuels based on anaerobic digestion of wet organic substrates, with CO₂ capture or no negative climate consequence.

Disclosure of invention

- 15 The above mentioned objects are achieved by the method according to the invention as defined by claim 1.

According to another aspect the invention concerns a device for performing the method as defined by claim 12.

Preferred embodiments are disclosed by dependent claims.

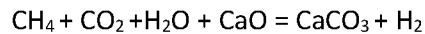
- 20 By "raw biogas" as discussed herein is understood a biogas from which Sulfur have been removed but in which the content of CO₂ is as originated from the anaerobic digester reactor, contrary to upgraded biogas which is essentially pure methane.

Addition of H₂ to the digestion process increases the ratio between bio-methane and CO₂, with CO₂ content potentially lower than 20%.

- 25 A main feature of the present invention is the conversion of the raw biogas with enhanced methane: CO₂ ratio directly in a sorption enhanced reforming (SER) process without prior separation of CO₂. This is achieved by dimensioning the reformer 70 to capture both CO₂ from the initial desulfurized raw biogas (CH₄ + CO₂) and the CO₂ formed in the reforming step (SE-SMR process).

Ca-looping has, to our knowledge, not been suggested as a method for CO₂ removal in relation to production of hydrogen from raw biogas or gases with considerable amounts of initial CO₂ in addition to methane.

The total SER-process with desulfurized raw biogas as feed gas is illustrated in the chemical reaction (not balanced) given below;



Two alternative embodiments of the present invention are illustrated below, namely (1) Biological conversion of CO₂ to CH₄ by the addition of H₂ from an SER-process in an AD reactor and (2) Direct conversion of desulfurized raw biogas (CH₄ + CO₂) to hydrogen in a SER reactor.

As illustrated and exemplified below the present invention provides in one embodiment a combined system for production of vehicle grade biomethane, and vehicle grade hydrogen, with the option of total CO₂ capture, from anaerobic digestion of organic waste.

Cost efficiency and sustainability are keyword and common denominators for the overall process.

Detailed description of the invention

Different embodiments of the invention are illustrated below with reference to the enclosed drawings, where:

Figure 1 is a schematic view of a first embodiment of the present invention, in which vehicle grade hydrogen is produced from anaerobic fermentation of biological material in a cost-efficient and sustainable manner.

Figure 2 is a schematic view of another embodiment of the present invention, in which vehicle grade hydrogen and vehicle grade biomethane is produced from anaerobic fermentation of biological material in a cost-efficient and sustainable manner.

Figure 3 is a schematic view of a third embodiment of the present invention, in which vehicle grade hydrogen is produced from anaerobic fermentation of biological material in a cost-efficient and sustainable manner, while also producing electricity.

Figure 4 is a schematic view of a fourth embodiment of the present invention, in which vehicle grade hydrogen and vehicle grade biomethane is produced from anaerobic fermentation of biological material in a cost-efficient and sustainable manner, while also producing electricity.

Attention is drawn to fig. 1. An anaerobic digester 50 for biogas production is charged with a charge material 11 based on sewage sludge, domestic organic waste, animal or agricultural waste.

The produced raw biogas 12 is desulfurized 60, and the desulfurized, still raw biogas 13 (CH_4 + initial CO_2) is charged to a reformer 70 together with water 14 in the form of steam for a sorption enhanced reforming (SER) process. A substantially pure hydrogen 15 gas leaves the reformer unit. In the reformer unit, one part 15b of which typically being returned to the digester 50 to enhance the methane yield while another and typically larger part 15a is subjected to purification in a hydrogen-purifier 100. The hydrogen purification may typically be performed as a pressure swing adsorption process. The purified hydrogen 16 leaving the purifier is of vehicle grade.

While the step of desulfurization 60 is a step commonly used in such processes and not inventive as such, it is a step which for practical chemical purposes will rarely or never be omitted.

10 The process of reforming and CO_2 capture in the reformer 70 involves a reaction between fuel (CH_4), water (steam), CO_2 (both from the original biogas and from the SER process) and CaO as a CO_2 absorber, a process in which CaO is converted to CaCO_3 in an exothermic reaction known per se.

15 The off-gas 17 from the hydrogen purification unit 100 is mixed with raw biogas 13c, which may be a partial flow of biogas flow 13, and charged to a burner 90 for production of the necessary heat (850 to 900 °C) for regeneration of CaCO_3 to CaO in a CaO regenerator 80, in an endothermic process. The CO_2 (100%) flow 23 produced in the reformer 70 and released in regenerator 80, may be used or stored (sequestration). The burner 90 is also charged with an oxygen containing gas 18, typically air.

20 The CO_2 in the exhaust from the burner 90 would have no climatic consequence since the fuel source is of biogenic origin. In addition, the CO_2 flow captured as flow 23 has a "negative" CO_2 climate impact, if this flow of CO_2 is stored or used.

25 Flow 19 is a flow from the reformer 70 to the regenerator 80 of solid CaCO_3 , resulting from CaO having absorbed CO_2 , while flow 20 is a flow of solid CaO , converted back from CaCO_3 , from the regenerator 80 back to the reformer 70. This Ca-looping process is well known as such, but not in the context here presented.

Attention is now directed to fig. 2. Most of the components and flows of figure 2 are the same as the ones in figure 1 and are numbered equally. The process according to Figure 2, however, has the additional ability of producing vehicle grade biogas. A first part 13a of the desulfurized biogas 13 is charged to the reformer 70 like in Figure 1 and treated accordingly. A second part of the biogas 13b from the desulfurization unit 60 is charged to a CO_2 separation unit 110. The CO_2 -separation is normally carried out by water scrubbing, physical or chemical absorption using

organic solvents, pressure swing adsorption, or by permeation using membranes, depending on the location and size of the system. The bio methane 24 discharged from CO₂ separation unit 110 may be said to be of natural gas quality or of vehicle grade and hence used for such purposes.

5 The CO₂ 25 released from the CO₂ separation unit 110 may be stored or used, if the method applied makes this economically feasible. This is however usually not the case. Regardless of the method used, the biogenic origin of the fuel source would result in no climatic consequence. The purity of the CO₂ 25 released from the CO₂ separation unit 110 depends on the type and nature of this unit.

10 Attention is now directed to fig. 3. Most of the components of figure 3 are the same as the ones in figure 1 and are equally numbered. The process according to Figure 3, however, has the additional ability of producing electricity and high temperature heat due to the presence of a solid oxide fuel cell (SOFC) integrated in the process and equipment. Thus, the substantially pure hydrogen discharged from the reformer, is typically divided into three substreams, namely substream 15a which is charged to the SOFC, substream 15b which is recycled to the digester 50 (like in Fig. 1 and
15 2) and substream 15c which, when present, is used as a fuel for a heater 95.

The hydrogen substream 15a is partially used to produce electricity in the SOFC while another part of the hydrogen flow 16' leaves the SOFC for further upgrading in a hydrogen purifier 100' which may or may not be similar to the unit 100 shown in Figures 1 and 2 to obtain vehicle grade hydrogen 26. The electricity may be used internally or externally or both.

20 The high temperature exhaust gas of the SOFC is used to heat the regenerator 80, but may typically need some assistance since the temperature needed in the regenerator 80 is 850 to 900 °C. This temperature may be reached (without any assistance) if ceramic interconnects are used in the SOFC system.

25 In practice, however, the temperature of the exhaust gas (830 °C, Megel et. al 2013) is too low to effectively provide a temperature in the regenerator at which the CaCO₃ is converted to CaO for further use. A dedicated system, to elevate the temperature of the exhaust gas in a temperature increasing cell/heating device, would thus be necessary.

The heat integration between the SOFC 120 and the Regenerator 80, via the heater 95, is in figure 3 provided by a closed heat loop 21', 22', however other options are possible.

30 The heat transfers medium of the heat loop 21', 22' in fig. 3, can be different gases, such as for example; Hydrogen, CO₂, air, helium, water vapor, different gas mixtures or fluids such as; mineral oils, hydrocarbons and different types of molten salts. The heat of the heat loop in figure 3, leaving

the SOFC system, is typically about 830 °C. The heat of this heat loop is enhanced in the heater 95 to at least 850 °C, more preferable at least 950 °C and most preferred at least 1000 °C, in order to meet the temperature regeneration requirement in the regenerator 80 where CaCO₃ is converted to CaO while releasing CO₂. While the heater 95 may be heated in different ways, one convenient way is the use of a partial flow of hydrogen 15c from the reformer. Another option for fuel to 95 would be raw biogas 13c. Whether the fuel for the heater has the form of raw biogas 13c according to figures 1, 2, or 4 or hydrogen 15c according to figure 3, the heat delivered by the burner 90 or by the heater 95 is at least partially provided, directly or indirectly, by the desulfurized raw bio-gas to be upgraded. The SOFC exhaust air 27 provides the oxygen for the heating process in 95. The CO₂ in the exhaust outlet 28 from the heating device would be climate neutral because of the biogenic origin of the fuel used. If required, additional air (not shown) may be supplied to the heater 95.

Attention is now directed to figure 4. Most of the components in figure 4 are the same as in figure 3. The process of Figure 4, however, has the additional ability of allowing production of vehicle grade biogas, in a manner similar to the difference between figure 1 and Figure 2.

Thus, according to Figure 4, the flow of desulfurized biogas 13 is split into a first flow 13a which is charged to the reformer 70 while the other flow 13b is charged to a CO₂ separation unit 110 to be separated into vehicle grade biomethane 24 and CO₂ 25. The treatment of the first flow 13a is as described above in relation to Figure 1, while the other flow 13b is subjected to a treatment as generally described in relation to Figure 2.

With regard to the SOFC and the processes involved therein, there is no difference between the embodiments of Figure 3 and 4. The same is to be said about the closed loop heat exchange 21', 22' including the use of the heater 95.

It is to be understood that the processes according to Figures 2 and 4 allows a split of the desulfurized raw biogas into a first flow 13a and a second flow 13b, covering a ratio between the two from 5:95 to 100:0. Thus, if demand is high for vehicle grade biomethane and low for vehicle grade hydrogen and electricity, the flow 13a would be reduced so as to basically just cover the need for hydrogen in the digester. On the other hand, if the demand for vehicle grade biomethane is low, the flow 13b could basically be cut off, rendering the embodiment of Figure 2 temporarily identical to the embodiment of Figure 1 and/ or rendering the embodiment of Figure 4 temporarily identical to the embodiment of Figure 3.

The general concept of the present invention is a method for the manufacture of vehicle grade fuels from biological materials in a cost-efficient and sustainable manner, involving a minimum of

steps. There is a versatility in the method in the sense that vehicle grade biomethane and vehicle grade hydrogen may be produced at a flexible mutual ratio, as well as flexible amounts of electricity.

Embodiments

- 5 While not representing the core of the present invention, a step of desulfurization 60 is typically conducted upstream of the step of sorption enhanced reforming 70.

As explained in relation to the drawings, a partial flow of desulfurized biogas 13 is according some embodiments subjected to treatment in a CO₂ separation unit 110 thereby providing one discharge flow of vehicle grade biomethane 24 and one discharge flow 25 containing CO₂.

- 10 The CO₂ separation unit 110 is typically one using a principle for separation selected among water scrubbing, physical or chemical absorption using organic solvents, pressure swing adsorption, and permeation using membranes.

- According to at least some embodiments the heat required for regenerating CaO is provided by burning a gas containing a partial flow of desulfurized biogas. In some embodiments heat for
15 regeneration of CaO may also be provided in part from a solid oxide fuel cell 120 charged with hydrogen 15a from the sorption enhanced reforming step 70. Additional heat may in case be provided by a heater 95 charged with hydrogen 15c discharged from the sorption enhanced reforming step 70. In other embodiments the fuel cell 120 may be charged with raw biogas or a combination of hydrogen and raw biogas.

- 20 In some embodiments a heat medium 21', 22' is circulated in a closed loop between at least the solid oxide fuel cell 120, the heater 95 and the regenerator 80. The addition of hydrogen to the digestion process may be arranged at least in part as a recycle hydrogen flow 15b from the reforming step 70.

- In a number of embodiments the raw biogas to be upgraded is produced in a digestion process
25 based on digestion of a raw material selected among one or more of sewage, sludge, municipal waste, domestic waste, animal waste, and agricultural waste.

List of references

- 11 feed to digester 50
 12 biogas from digester 50
 13 desulfurized biogas to reformer 70
 5 13a partial flow desulfurized biogas to reformer 70
 13b desulfurized biogas to CO₂ separator 110
 13c raw, desulfurized biogas to heater 95/ burner 90
 14 water to reformer 70
 15 substantially pure hydrogen from reformer 70
 10 15a partial flow hydrogen to reformer 70
 15b recycle partial flow of hydrogen to digester 50
 15c partial hydrogen flow to heater 95
 16 vehicle grade hydrogen from hydrogen purifier 100
 16' Subst pure hydrogen from SOFC 120
 15 17 off gas from hydrogen purifier 100
 18 air inlet to burner 90
 19 CaCO₃ from reformer 70 to regenerator 80
 20 CaO from regenerator 80 to reformer 70
 21 hot gas from burner 90 to regenerator 80
 20 21' hot gas from heater 95 to regenerator 80
 22 off gas from regenerator 80
 22' off gas from regenerator to SOFC 120 in closed loop
 23 CO₂ from regenerator 80
 24 vehicle grade bio-gas from CO₂ separator 110
 25 25 CO₂ from CO₂ separator 110
 26 Vehicle grade hydrogen from 100'
 27 Exhaust from SOFC 120
 28 Exhaust from heater 95

 30 50 digester
 60 desulfurizer
 70 reformer
 80 regenerator
 90 burner
 35 95 heater
 100 hydrogen purifier
 100' hydrogen purifier
 110 CO₂ separator
 120 solid oxide fuel cell
 40

Claims

1. Method for upgrading biogas from anaerobic fermentation of biological material under production of energy rich gases selected among methane (24) and hydrogen (16, 26) or a combination thereof, comprising addition of hydrogen gas to the fermentation step (50) to
5 enhance the methane:CO₂ ratio in the raw biogas (12) produced and subjecting the raw biogas to a step of desulfurization to the extent required **characterized in** further comprising subjecting at least a part of the desulfurized raw biogas to a step of sorption enhanced reforming (70) without prior separation of CO₂, using CaO as an absorbent to capture CO₂ from the raw biogas as well as CO₂ released in the reforming reaction, regenerating CaO in an endothermic reaction (80) using
10 heat at least partially provided, directly or indirectly, by the bio-gas to be upgraded, thereby producing substantially pure hydrogen (15) and substantially pure CO₂ (23).
2. Method as claimed in claim 1, wherein a step of desulfurization (60) is conducted upstream of the step of sorption enhanced reforming (70).
3. Method as claimed in any one of claims 1 - 2, wherein a partial flow of desulfurized raw biogas
15 (13) is subjected to treatment in a CO₂ separation unit (110) thereby providing one discharge flow of vehicle grade biomethane (24) and one discharge flow (25) containing CO₂.
4. Method as claimed in claim 4 wherein the CO₂ separation unit (110) is one using a principle for separation selected among water scrubbing, physical or chemical absorption using organic solvents, pressure swing adsorption, and permeation using membranes.
- 20 5. Method as claimed in any one of the preceding claims wherein the substantially pure H₂ (15) is subjected to a step (100) of further purification to produce vehicle grade H₂ (16).
6. Method as claimed in any one of the preceding claims, wherein the heat required for regenerating CaO is provided by burning a gas containing a partial flow of desulfurized raw biogas (13c).
- 25 7. Method as claimed in any one of claims 1 – 5, wherein the heat for regeneration of CaO is provided in part from a solid oxide fuel cell (120) charged by hydrogen (15a) from the sorption enhanced reforming step (70), by desulfurized raw biogas 13c or a combination thereof.
8. Method as claimed in claims 7, wherein additional heat for regeneration of Ca O is provided by a heater (95) charged with hydrogen (15c) discharged from the sorption enhanced reforming step
30 (70).

9. Method as claimed in one of claims 7 or 8, wherein a heat medium (21', 22') is circulated in a closed loop between at least the solid oxide fuel cell (120), the heater (95) and the regenerator (80).
10. Method as claimed in any one of claims 1-9 wherein the raw biogas to be upgraded is produced in a digestion process (50) based on digestion of a raw material selected among one or more of sewage, sludge, municipal waste, domestic waste, animal waste, and agricultural waste.
11. Method as claimed in claim 9 wherein the addition of hydrogen to the digestion process is arranged at least in part as a recycle hydrogen flow (15b) from the reforming step (70)
12. Device for upgrading biogas from anaerobic fermentation of biological material under production of energy rich gases, selected among methane (24) and hydrogen (16) or a combination thereof, comprising a digester (50), a device (60) for desulfurization as well as a reformer unit (70, 80) **characterized in** that the reforming unit (70, 80) is arranged downstream of the digester (50) and the device for desulfurization, for reforming according to sorption enhanced reforming principle based on Ca-looping to provide a reforming which is inherently integrated with carbon capture and subsequent purification of the hydrogen (15) with a hydrogen purification unit (100, 100').
13. Device as claimed in claim 12, wherein a solid oxide fuel cell (120) is arranged to receive the at least part of the gas discharged from the reformer, is subjected to purification in a hydrogen purification unit (100).
14. Device as claimed in claim 12, wherein a solid oxide fuel cell (120) is arranged to receive the at least part of the gas discharged from the reformer, is charged to a solid oxide fuel cell for production of electric energy and heat.
15. Device as claimed in claim 12, wherein part of the gas discharged from the desulfurizer is charged to a CO₂ separation unit to split the gas flow into a vehicle grade biomethane (24) and pure CO₂ (25).

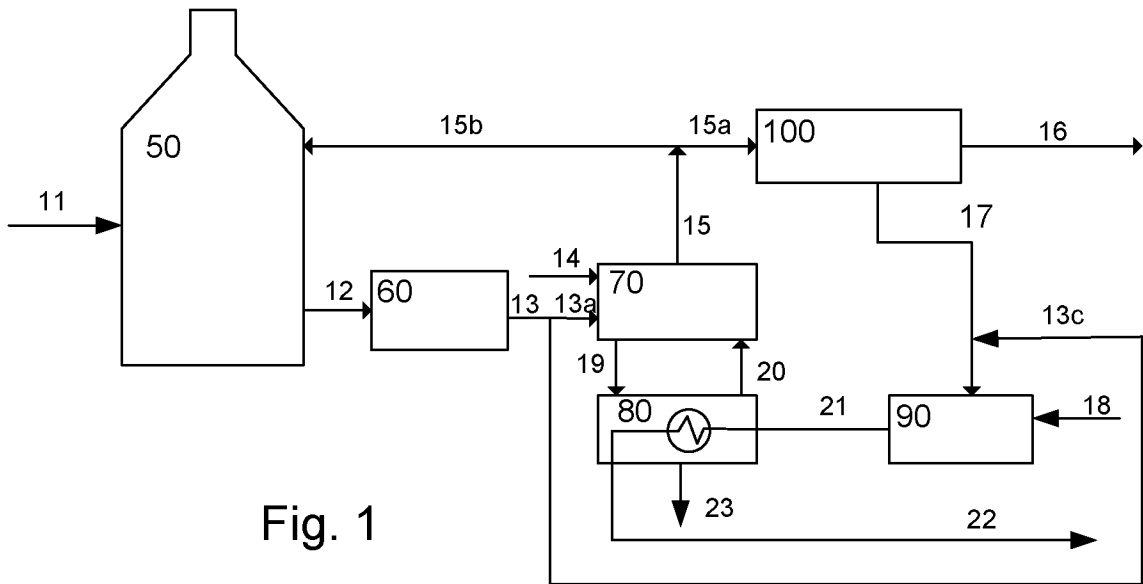


Fig. 1

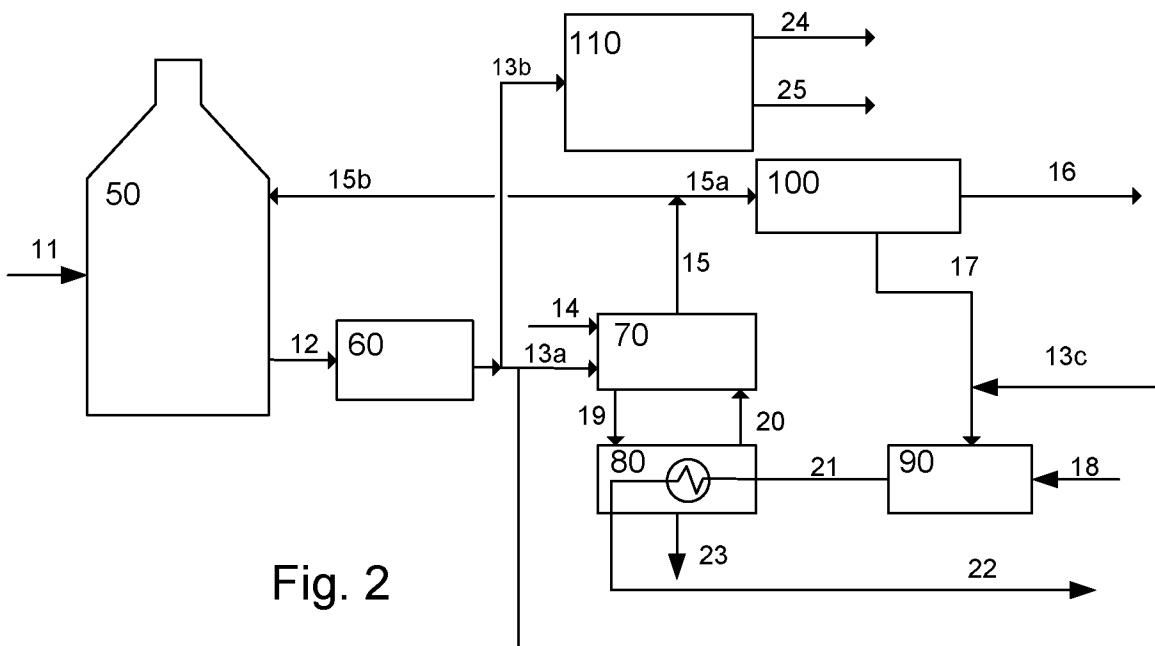


Fig. 2

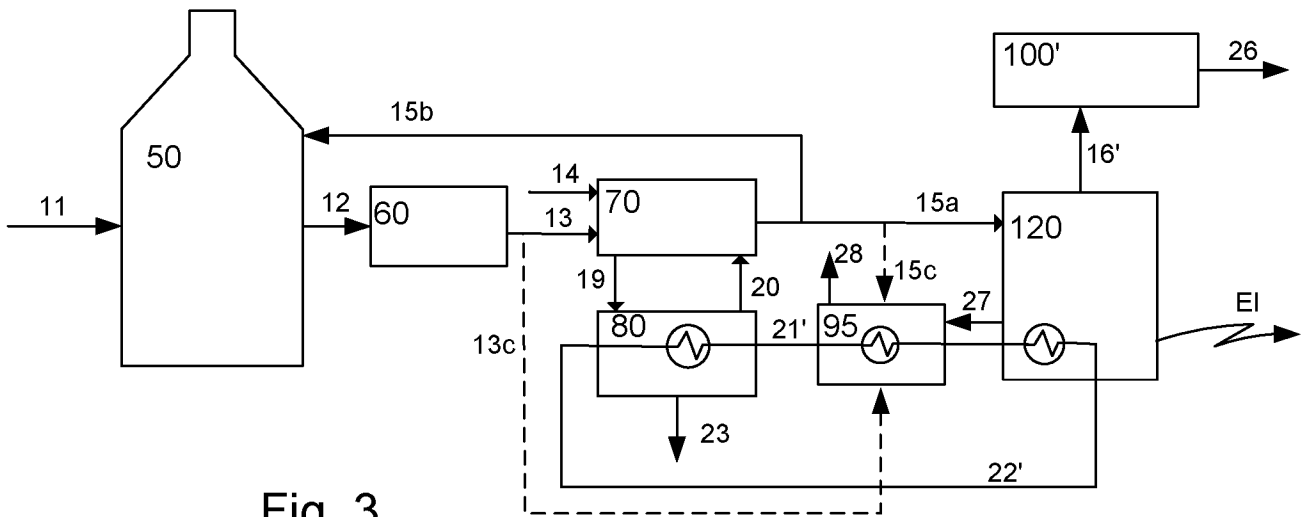


Fig. 3

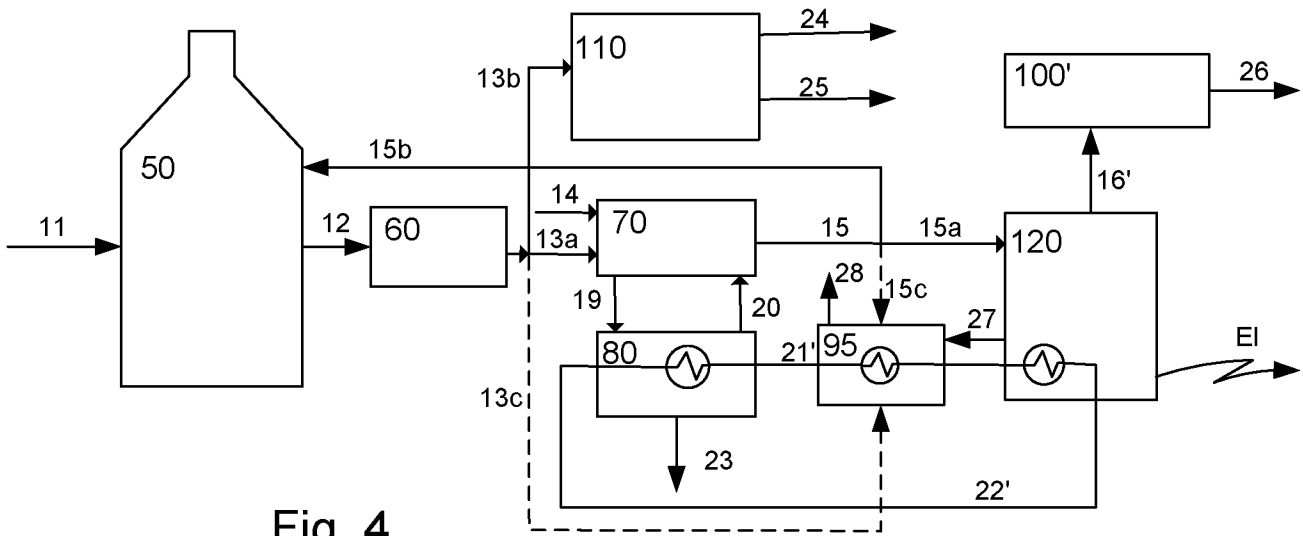


Fig. 4

INTERNATIONAL SEARCH REPORT

International application No
PCT/N02017/050185

A. CLASSIFICATION OF SUBJECT MATTER
INV. C01B3/34 C12P5/02
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)
C01B C12P

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 practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 105 084 312 A (UNIV ZHEJIANG) 25 November 2015 (2015-11-25) the whole document	12
Y	US 2007/298478 A1 (OFFERMAN JOHN D [US] ET AL) 27 December 2007 (2007-12-27) paragraph [0033] - paragraphs [0038], [0047]; figure 2 paragraphs [0052], [0061]; example 1	1-6, 10-12,15
Y	US 2005/229489 A1 (BAVARIAN FARSHAD A [US] ET AL) 20 October 2005 (2005-10-20) paragraphs [0029], [0038] paragraphs [0052], [0062] - paragraphs [0076], [0084]	1-6, 10-12,15
A	WO 2014/200357 A1 (ZEG POWER AS [NO]) 18 December 2014 (2014-12-18) figures 1,2	7-9,13, 14

Further documents are listed in the continuation of Box C.

See patent family annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
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- "O" document referring to an oral disclosure, use, exhibition or other means
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- "&" document member of the same patent family

Date of the actual completion of the international search 5 September 2017	Date of mailing of the international search report 13/09/2017
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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 Cristescu, Ioana
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No PCT/N02017/050185

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